Pilot Testing of Mercury Oxidation Catalysts for Upstream of Wet FGD Systems

Quarterly Technical Progress Report

January 1, 2004 – March 31, 2004

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ABSTRACT

This document summarizes progress on Cooperative Agreement DE-FC26-01NT41185, "Pilot Testing of Mercury Oxidation Catalysts for Upstream of Wet FGD Systems," during the time-period January 1, 2004 through March 31, 2004. The objective of this project is to demonstrate at pilot scale the use of solid honeycomb catalysts to promote the oxidation of elemental mercury in the flue gas from coal combustion. The project is being funded by the U.S. DOE National Energy Technology Laboratory under Cooperative Agreement DE-FC26-01NT41185. EPRI, Great River Energy (GRE), and City Public Service (CPS) of San Antonio are project co-funders. URS Group is the prime contractor.

The mercury control process under development uses catalyst materials applied to honeycomb substrates to promote the oxidation of elemental mercury in the flue gas from coal-fired power plants that have wet lime or limestone flue gas desulfurization (FGD) systems. Oxidized mercury is removed in the wet FGD absorbers and co-precipitates with the byproducts from the FGD system. The current project is testing previously identified catalyst materials at a larger, pilot scale and in a commercial form, to provide engineering data for future full-scale designs. The pilot-scale tests will continue for approximately 14 months or longer at each of two sites to provide longer-term catalyst life data.

This is the tenth full reporting period for the subject Cooperative Agreement. During this period, project efforts included continued operation of the first pilot unit at the GRE Coal Creek site with all four catalysts in service and sonic horns installed for on-line catalyst cleaning. During the quarter, one catalyst activity measurement trip was completed, and catalyst pressure drop was monitored with the sonic horns continuing in operation. For the second pilot unit at CPS' Spruce Plant, the catalyst pilot unit continued in operation throughout the quarter. One catalyst activity measurement trip was conducted, in mid-February. Also, a measurement trip in early January measured mercury oxidation and removal across the fabric filter prior to rebagging, which began in mid-January. These measurements were made to determine the effect of new versus aged bags in the fabric filter on flue gas elemental mercury concentrations at the catalyst pilot inlet. Finally, results became available from the December mercury SCEM relative accuracy tests conducted at Spruce plant.

This technical progress report details available results from these efforts at both sites.

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INTRODUCTION

This document is the quarterly Technical Progress Report for the project "Pilot Testing of Mercury Oxidation Catalysts for Upstream of Wet FGD Systems," for the time-period January 1, 2004 through March 31, 2004. The objective of this project is to demonstrate at pilot scale the use of solid honeycomb catalysts to promote the oxidation of elemental mercury in the flue gas from coal combustion. The project is being funded by the U.S. DOE National Energy Technology Laboratory under Cooperative Agreement DE-FC26-01NT41185. EPRI, Great River Energy (GRE) and City Public Service (CPS) of San Antonio are project co-funders. URS Group is the prime contractor.

The mercury control process under development uses catalyst materials applied to honeycomb substrates to promote the oxidation of elemental mercury in the flue gas from coal-fired power plants that have wet lime or limestone flue gas desulfurization (FGD) systems. The oxidizing species are already present in the flue gas, and may include chlorine, hydrochloric acid (HCl) and/or other species. Oxidized mercury is removed in the wet FGD absorbers and co-precipitates with the byproducts from the FGD system. The objective of this project is to test previously identified effective catalyst materials at a larger scale and in a commercial form to provide engineering data for future full-scale designs. The pilot-scale tests will continue for 14 months or longer at each of two sites to provide longer-term catalyst life data. After successful completion of the project, it is expected that sufficient full-scale test data will be available to design and implement demonstration-scale or commercial-scale installations of the catalytic mercury oxidation technology.

The two utility team members are providing co-funding, technical input, and host sites for testing. GRE is providing the first test site at their Coal Creek Station (CCS), which fires a North Dakota lignite, and CPS is providing the second site at their J.K. Spruce Plant, which fires a Powder River Basin (PRB) subbituminous coal. These two host sites each have existing wet FGD systems downstream of high-efficiency particulate control devices, an ESP at CCS and a reverse-gas fabric filter (baghouse) at Spruce**.**

The remainder of this report is divided into five sections: an Executive Summary followed by a section that describes Experimental procedures, then sections for Results and Discussion, Conclusions, and References.

EXECUTIVE SUMMARY

Summary of Progress

The current reporting period, January 1, 2004 through March 31, 2004, is the tenth full technical progress reporting period for the project. Efforts over the current period included continued operation of the first mercury oxidation catalyst pilot unit at the CCS site with all four catalysts installed and sonic horns in operation for on-line catalyst cleaning, and continued operation with all four catalysts installed in the second pilot unit at CPS' Spruce plant (but no sonic horns).

The pilot unit at CCS is installed at the outlet of an induced draft fan and downstream of the cold-side electrostatic precipitator on Unit 1. An SCR catalyst and a palladium-based catalyst (Pd #1) have been in operation since October 3, 2002. A subbituminous ash-based catalyst, SBA #5, was placed in service the first week in December 2003. The fourth, Carbon #6 (C #6) catalyst was installed and placed in service on June 5, 2003. During the current quarter, one set of catalyst activity measurements were made at the CCS site, and the pilot unit was monitored from off site to observe catalyst pressure drop values.

After ten months of operation with sonic horns in service for on-line catalyst cleaning, they appear to be effective in limiting fly ash buildup in the horizontal gas flow catalysts for three of the four catalysts. However, this observation is confounded by apparent measurement problems with the catalyst pressure drop transducers. During the quarter, the signals from the four catalyst transducers were very noisy, an effect thought to be due to moisture condensation in the tubing to the transducers during cold weather operation. For the C #6, Pd #1, and SCR catalysts, the pressure drop the average pressure drop values appear to remain below 1 in. H_2O. For the fourth catalyst, SBA #5, after several fluctuations the pressure drop was up to about 4 in. H_2O at the end of the quarter. This suggests that the sonic energy level is not sufficient to prevent fly ash buildup across this catalyst, or it may be a result of damage to the catalyst substrate from the current levels of sonic energy.

A catalyst activity measurement trip conducted in February showed 88% Hg^0 oxidation for the C #6 catalyst, about 66% oxidation for the Pd #1 but significantly lower activity (<30% oxidation) for the SCR and SBA #5 catalysts, as measured with a mercury SCEM.

At CPS' Spruce Plant catalyst activity results were measured in February. These measurements showed that the fabric filter outlet flue gas mercury content is still highly oxidized (~75% or greater), in spite of 11 of the 14 compartments in the West fabric filter having been rebagged. The fabric filter was being rebagged in January/February, replacing the 11-year-old bags. It was hoped that new bags would reduce the observed mercury oxidation across the fabric filter. Based on the February results, the new bags did not markedly impact mercury oxidation across the fabric filter. The relatively low inlet elemental mercury concentrations to the pilot unit (about 2.5 $\mu g/Nm^3$) make it difficult to quantify catalyst oxidation activity. The catalyst activity results from this trip indicate approximately 75% elemental mercury oxidation across the gold and SCR catalysts, 60% oxidation across the Pd #1 catalyst, and less than 20% oxidation across the C #6 catalyst. The latter result is surprising, given that the C #6 catalyst is performing well at Coal

Creek. The next measurement trip will be in early April, and will be used to confirm these results.

An advantage of having a fabric filter rather than an ESP upstream of the catalyst pilot unit is that there has been no tendency for fly ash buildup in the catalyst chambers. No sonic horns have been installed on the pilot unit at Spruce, and the pressure drop across all four catalysts remains below 0.3 in. H_2O.

Also during the quarter, the results from mercury SCEM relative accuracy tests using the Ontario Hydro method and other gas characterization tests, conducted at Spruce during December 2003, became available. The results from this December measurement trip are presented report.

No subcontracts were issued during the current reporting period.

Problems Encountered

There were no significant new problems encountered during the reporting period, other than the technical issues described in Section 4 of this report and mentioned above.

Plans for Next Reporting Period

During the next reporting period (April 1 through June 30, 2004), a final intensive flue gas sampling trip will occur at the end of the long-term catalyst evaluation period at CCS (May 2004), after which the pilot unit will be shut down. One routine sampling trip will be conducted in April to evaluate catalyst activity at CCS. The original project schedule called for pilot unit operation to end after 14 months in service (~December 2003). However, the schedule was extended because the C #6 catalyst has only been operation since June 2003, and more operating time is needed to be able to predict its life. The operating period was also extended to allow testing with a pilot-scale wet scrubber as part of another DOE-funded project (DE-FC26-04NT41992), to determine how effectively the catalytically oxidized mercury will be scrubbed.

Operation of a second oxidation catalyst pilot unit, at CPS' Spruce Plant, will continue with all four catalysts installed. Routine sampling trips will be conducted to evaluate catalyst activity. Gas measurements will be made with the mercury SCEM to evaluate the impacts of the host unit fabric filter bag change on mercury oxidation at the catalyst pilot inlet. An intensive gas characterization effort should occur in June or early July.

Prospects for Future Progress

During the next reporting period (July 1 through September 30, 2004), no testing is scheduled at the CCS site, as the pilot unit will have been shut down. At the second site, CPS' Spruce Plant, pilot unit operation should continue until the end of calendar year 2004, and catalyst activity will be evaluated for elemental mercury oxidation activity through routine (~monthly to bimonthly) evaluation trips. Intensive gas characterization efforts should occur at the end of the test period, in December 2004.

EXPERIMENTAL

The work described in this technical progress report was conducted using two different experimental apparatuses. One is an elemental mercury catalyst oxidation pilot unit (8000 acfm of flue gas treated) located at GRE's CCS Station in North Dakota. A second, nearly identical pilot unit is located at CPS' Spruce Plant. Each pilot unit has four separate compartments that allow four different catalysts to treat flue gas from downstream of the host plant's particulate control device and upstream of its FGD system. Details of the pilot unit design, construction, catalyst preparation and pilot unit operation have been discussed in previous quarterly technical progress reports[1,2,3,4]. The activity of these catalysts is being determined by measuring the change in elemental mercury concentration across each catalyst, while ensuring that the total mercury concentrations do not change significantly across the catalyst. These measurements are primarily being conducted using a mercury semi-continuous emissions monitor (SCEM) developed with funding from EPRI. The analyzer has been described in a previous report[5]. Periodically, the analyzer results are being verified by conducting manual flue gas sampling efforts in parallel across each catalyst chamber by the Ontario Hydro method.

The second experimental apparatus is a bench-scale test unit that is used to evaluate the activity of candidate catalyst cores under simulated flue gas conditions. However, no bench-scale tests were conducted during the current quarter. The bench-scale catalyst oxidation test apparatus was previously described in quarterly technical progress reports[3,4].

RESULTS AND DISCUSSION

This section provides details of technical results for the current reporting period, January 1, 2004 through March 31, 2004. The technical results presented include a discussion of the data from the first pilot unit at GRE's CCS and from the second pilot unit at CPS' Spruce Plant.

Pilot Unit Operation at CCS

Background

As described in the previous quarterly reports, the first pilot unit was started up at CCS with the SCR and Pd #1 catalysts the first week of October 2002. The other two catalysts (SBA #5 and C #6) were not yet available. Initial catalyst activity measurements, made using the EPRI mercury SCEM, showed over 90% oxidation of elemental mercury across the Pd#1 catalyst, as was expected based on previous laboratory and field test results. The SCR catalyst results showed lower oxidation, in the range of 60 to 70% oxidation of elemental mercury across the catalyst. Throughout this report, elemental mercury oxidation percentages across catalysts are reported based on the measured drop in elemental mercury concentration across the catalyst, and do not just reflect the total flue gas mercury oxidation percentage at the catalyst outlet.

In December 2002, the third catalyst, SBA #5, was installed and measurement results showed a marked decrease in activity for both the Pd #1 and SCR catalysts. Follow-up testing in January determined that the catalyst surfaces were becoming plugged due to buildup of fly ash, in spite of the catalyst being installed downstream of a high-efficiency ESP. This was confirmed by tracking pressure drop increases across the catalysts and by opening and physically inspecting the catalyst chambers to observe and clean out the fly ash buildup.

It appeared that mechanical catalyst cleaning would be needed on the pilot unit. Both air soot blowers and sonic horns were considered. It was decided that a sonic horn would be the easiest field retrofit and would offer a good probability of success. A small, 17-inch horn produced by Analytec Corporation of Pagosa Springs, Colorado appeared to be the best solution based on price, availability, and probability of success. During the last week of March 2003, the sonic horn was installed on the Pd #1 catalyst box to provide an occasional pulse of acoustic energy to the catalysts to dislodge accumulated particulate matter. The horn was installed on the top wall of the catalyst housing inlet transition, approximately 1.5 feet upstream of the first catalyst module. The horn sounds for 10 seconds every half hour. At the time the sonic horn was installed, the catalyst housing was opened and the Pd #1 catalyst modules were cleaned.

The pilot unit was placed back in service on March 27, and the horn proved to be effective at controlling pressure drop across the Pd #1 catalyst. A catalyst activity measurement trip was conducted the week of April 23. While the Pd #1 results were confounded by apparent mercury adsorption across the catalyst (i.e., a portion of the drop in elemental mercury concentration across the Pd #1 could be due to adsorption rather than oxidation) they otherwise showed high (~90%) elemental mercury oxidation across the catalyst. Based on the relatively high activity and low pressure drop values for Pd #1, sonic horns were installed on the other three boxes by CCS plant personnel the first week of June 2003.

Catalyst Pressure Drop Results

With the horns in service, the pressure drops across three of the four catalysts have stayed low. The pressure drop values since June 5 are plotted in Figure 1. At the beginning of the quarter, the C #6 pressure drop was about 0.4 in. H_2O, and the SCR catalyst (larger pitch and 1500 acfm flow rate) pressure drop was about 0.2 in. H_2O. The signal for the pressure drop across the Pd #1 catalyst became very noisy on about November 5, presumably due to water buildup in the tubing to the pressure drop transducer. The same thing happened with this transducer during cold weather operation the previous winter. However, the last "good" data on November 5 showed that the pressure drop across this catalyst remained low at 0.35 in. H_2O.

The SBA #5 pressure drop has continued to increase with time, to nearly 4 in. H_2O by the end of December, more than 10 times the initial pressure drop on June 5. It may be that there is a particle-to-particle attraction between the fly ash in the flue gas treated and the fly ash imbedded in the catalysts. Another possibility is that the sonic energy from the horn sounding in this compartment has damaged the honeycomb structure, thus causing a pressure drop increase due to blocked cells. This catalyst type is of lesser interest for future commercial applications, so regardless of the cause, the pressure drop increase across this catalyst chamber is not of great concern.

During the current quarter, the signals for pressure drop across all four catalysts became very noisy, again presumably due to water buildup in the tubing to the transducers during cold weather operation. In January, only the pressure drop transducers for the SCR and SBA #5 catalysts appeared to remain readable. The pressure drop across the SCR catalyst remained below 0.3 in H_2O and the pressure drop across the SBA #5 catalyst remained high at about 4 in. H_2O. After the end of January none of the four catalyst pressure drop values were readable.

Catalyst Activity Results

One catalyst activity measurement trip was made to CCS during the quarter, with all of the measurements being made on February 16. The results of the catalyst activity measurements (by SCEM) are shown in Table 1. The inlet flue gas mercury concentrations varied during the day, averaging about 13 $\mu g/Nm^3$ in the morning and 18 $\mu g/Nm^3$ of total mercury in the afternoon. At the higher inlet total mercury concentration, the observed pilot unit inlet mercury oxidation averaged 15%. As has been seen in results presented in the previous technical progress reports, all four catalysts appeared to be adsorbing a small amount of mercury from the inlet flue gas, ranging from 3% to 9% apparent adsorption. However, this small amount of mercury adsorption could be within measurement error given the fluctuating total inlet mercury concentrations seen over the course of the day.

The activity of the C #6 and Pd #1 catalysts remained relatively high, with 87 to 89% Hg^0 oxidation across the C #6 catalyst and 66% Hg^0 oxidation across the Pd #1. However, the measured activity for each was slightly lower than was measured last, in December. The activities of the SBA #5 and SCR catalysts continue to be lower than the C #6 and Pd #1 catalysts, in the range of about 20% to 30% Hg^0 oxidation. The activity of both catalysts dropped measurably since December.

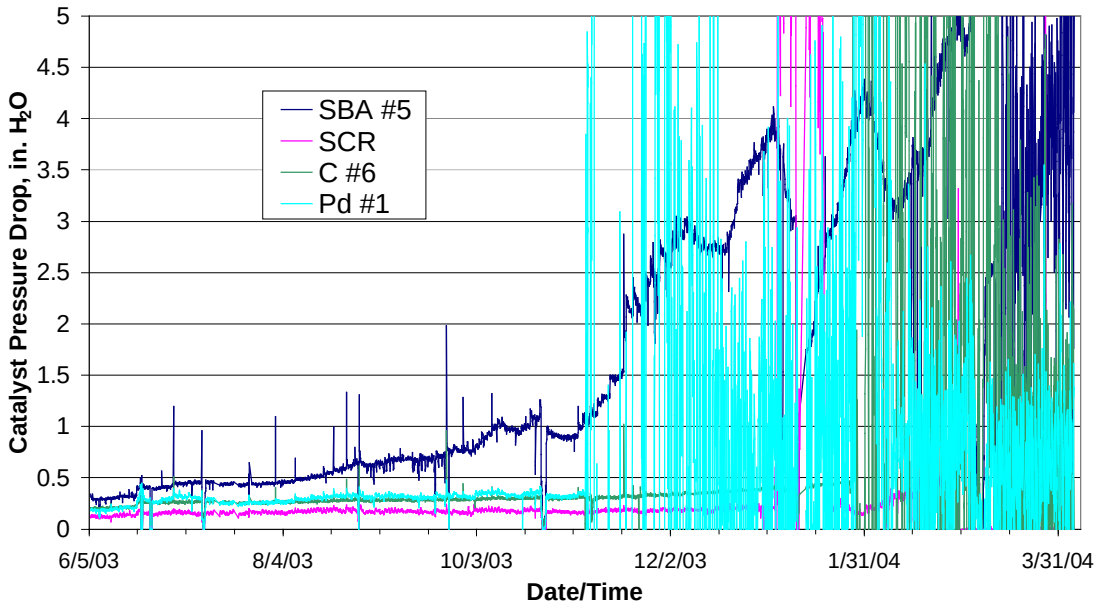

Figure 1. Pressure Drop Data for the Catalysts in Service at CCS through December

Table 1. Oxidation Catalyst Activity Results for CCS Pilot (measured by Hg SCEM)

Location	Total Hg (mg/Nm^3, corrected to 5% O_2)	Elemental Hg (mg/Nm^3, corrected to 5% O_2)	Apparent Total Hg Adsorption Across Catalyst, %	Apparent Hg^0 Oxidation Across Catalyst, %	Overall Hg Oxidation Percentage
Results from 2/18/04 (a.m.):					
Pilot Inlet	13.1	14.2	-	-	*
SBA #5 Outlet	12.2	10.3	6.6	28	16
C #6 Outlet	12.3	1.84	6.5	87	85
Results from 2/18/04 (p.m.):					
Pilot Inlet	17.6	14.9	-	-	15
SCR Outlet	17.0	10.9	3.1	27	36
Pd #1 Outlet	15.9	5.06	9.4	66	68
Pilot Inlet	-	12.1	-	-	-
SBA #5 Outlet	-	9.36	-	23	-
C #6 Outlet	-	1.36	-	89	-

*Value not calculated; observed mercury oxidation was less than 0% due to fluctuations in inlet total mercury concentrations during time period measurements were made.

The “clean catalyst” activity results for all four catalysts are plotted versus time in Figures 2 and 3. Some data points from late 2002 and early 2003, where the catalysts were obviously plugged with fly ash, have been edited from these plots. Activity results for the Pd #1 and C #6 catalysts are plotted in Figure 2 and activity results for SBA #5 and SCR catalysts in Figure 3. The plots in

Figure 2 show a general downward trend in the clean catalyst activity measurements for the two more active catalysts. More time in service will help quantify the change in activity versus time for these catalysts. The data plotted in Figure 3 show relatively "flat" activity performance for the SBA #5 and SCR catalysts over time since the sonic horns were installed last June, albeit at relatively low oxidation percentages.

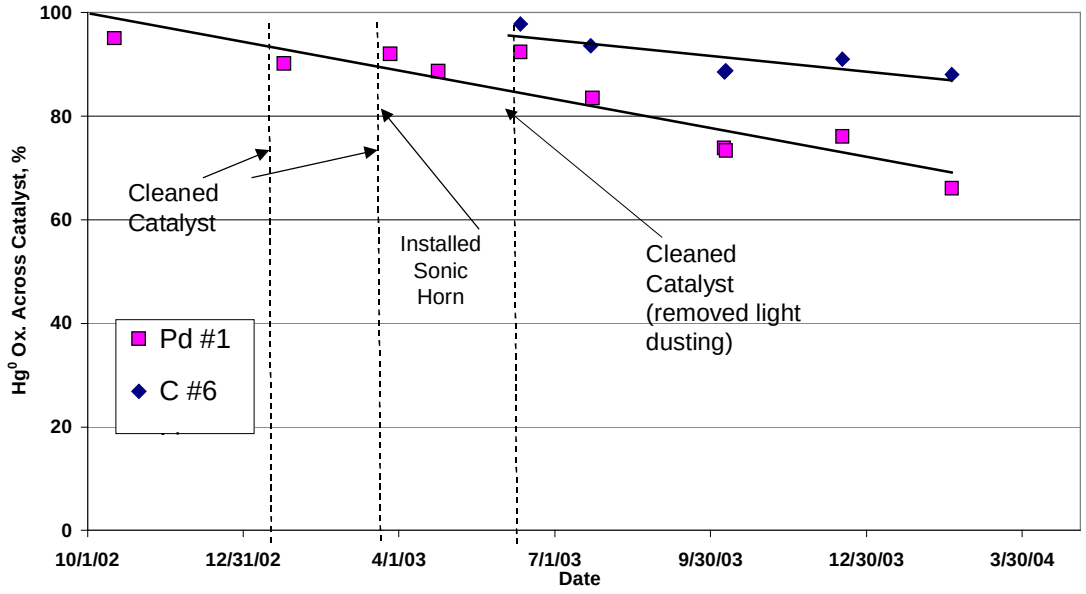

Figure 2. Activity for Hg^0 Oxidation versus Time for Pd #1 and C #6 Catalysts at CCS.

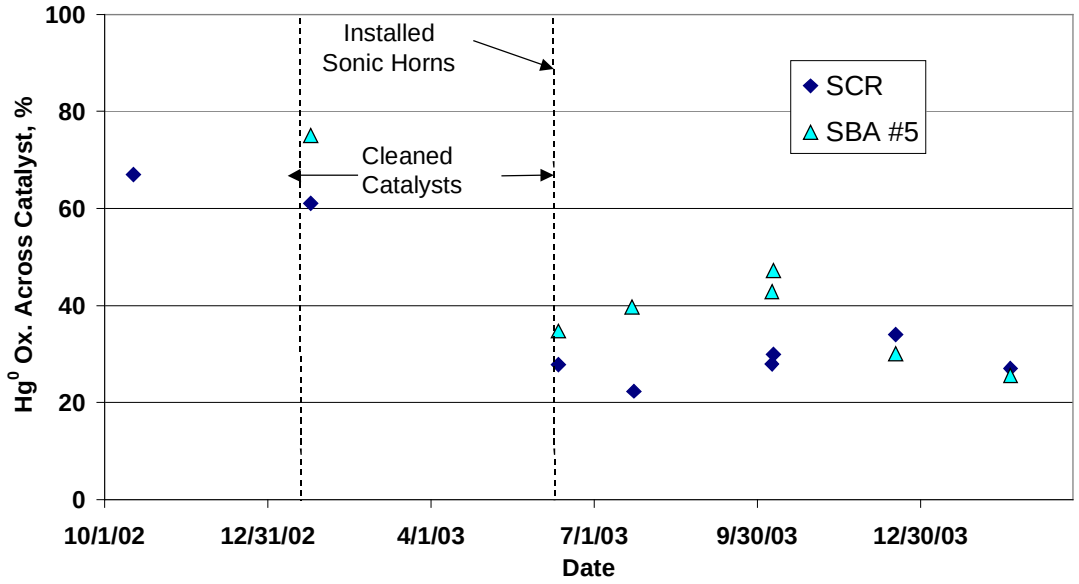

Figure 3. Activity for Hg^0 Oxidation versus Time for SCR and SBA #5 Catalysts at CCS.

Pilot Unit Operation at Spruce Plant

Background

The pilot unit was started up at Spruce Plant in late August 2003 and operated with the Pd #1 and Au catalysts installed for most of the month of September. The host unit came off line for a fall outage the evening of September 26, and the outage continued until October 27. The two remaining catalysts (SCR and C #6) were installed in the pilot unit and the pilot unit was restarted on November 13, about two weeks after the host unit came back on line. The unit has operated continuously with all four catalysts on line since then.

Pilot unit inlet and catalyst outlet mercury concentration data were collected at Spruce late in the previous quarter, the week of December 8. SCEM relative accuracy tests by the Ontario Hydro Method were conducted at the same time. These results were still being reduced and evaluated at the time the previous quarterly Technical Progress Report was prepared, so they are presented in this report.

Also, during the current quarter, flue gas mercury concentrations were measured at Spruce Plant by SCEM on two occasions. The week of January 5, two SCEMs were taken to the site and used to measure flue gas total mercury and elemental mercury concentrations at the fabric filter inlet and outlet, and at the wet FGD outlet locations on the host unit. These measurements were made to develop a baseline characterization of host unit flue gas mercury conditions prior to rebagging the fabric filter with new bags. The rebagging began on January 12. Routine catalyst activity measurements by Hg SCEM were made on February 13, after 11 of the 14 compartments in the west fabric filter (directly upstream of the catalyst pilot unit) had been rebagged.

Catalyst Pressure Drop Results

The pressure drop across the four catalyst chambers at Spruce remained nearly constant between 0.2 and 0.3 in H_20 from January 1 through March 31, the end of the current quarter. It does not appear that sonic horns will be required to prevent fly ash buildup, most likely because a high-efficiency reverse-gas fabric filter is used for particulate control at this site. The use of a fabric filter results in a low dust loading in the pilot unit inlet flue gas, and a dust loading that has less residual electrostatic charge than would flue gas downstream of an ESP.

Catalyst Activity Results

Two catalyst measurement trip results are presented in this report, with data from December 2003 and February 2004 being shown in Tables 2 and 3, respectively. As has been previously reported, the measurements at the pilot unit inlet showed high mercury oxidation percentages, with SCEM measurements showing 65% to 89% oxidized in December and February rather than the expected 20 to 30% oxidized mercury typical of PRB flue gases. This effect still appears to be an influence of the fabric filter used for particulate control at Spruce. The fabric filter operates at a low air-to-cloth ratio (less than 1.5 acfm/ft^2), and through early January 2004 had aged bags (11 years old) with a heavy permanent dust cake. The fabric filter was rebagged during the quarter. The gas measurements in February reflected operation with new bags in 11 of the 14 compartments in the west fabric filter that treats flue gas going to the catalyst pilot unit.

At that time, the partial rebagging had not markedly changed the mercury oxidation percentage at the fabric filter outlet, since the February 13 measurements at the catalyst pilot inlet still showed 76% oxidation.

Table 2. December Oxidation Catalyst Activity Results for Spruce Pilot (measured by Hg SCEM)

Location	Total Hg (μg/Nm^3, corrected to 5% O_2)	Elemental Hg (μg/Nm^3, corrected to 5% O_2)	Apparent Total Hg Adsorption Across Catalyst, %	Apparent Hg^0 Oxidation Across Catalyst, %	Overall Hg Oxidation Percentage
Results from 12/10/03:					
Pilot Inlet	10.4	3.60	-	-	65
C #6 Outlet	7.31	1.11	29	69	85
Au Outlet	5.85	0.86	43	76	85
Results from 12/11/03:					
Pilot Inlet	11.1	1.92	-	-	83
C #6 Outlet	13.5	1.16	0	40	91
Au Outlet	10.3	0.90	8	53	91
Results from 12/12/03:					
Pilot Inlet	13.0	1.47	-	-	89
Pd #1 Outlet	11.5	0.73	11	50	94
SCR Catalyst Outlet	15.1	0.66	0	55	96

Table 3. February 2004 Oxidation Catalyst Activity Results for Spruce Pilot (measured by Hg SCEM)

Location	Total Hg (μg/Nm^3, corrected to 5% O_2)	Elemental Hg (μg/Nm^3, corrected to 5% O_2)	Apparent Total Hg Adsorption Across Catalyst, %	Apparent Hg^0 Oxidation Across Catalyst, %	Overall Hg Oxidation Percentage
Results from 2/13/04:					
Pilot Inlet	10.3	2.45	-	-	76
Pd #1 Outlet	10.8	1.00	0	59	91
C #6 Outlet	10.8	2.01	0	18	81
Au Outlet	11.0	0.60	0	76	95
SCR Catalyst Outlet	11.8	0.61	0	75	95

In general, the catalyst activity results show quite a bit of variability over time, and the mercury oxidation percentages are lower than expected. Particularly in the December results, there are marked differences between the activity measured for the C #6 and Au catalysts on consecutive days, December 10 and December 11. All of these results are plotted versus time in Figure 4, including earlier results for the Pd #1 and Au catalysts from the initial month of operation in September 03.

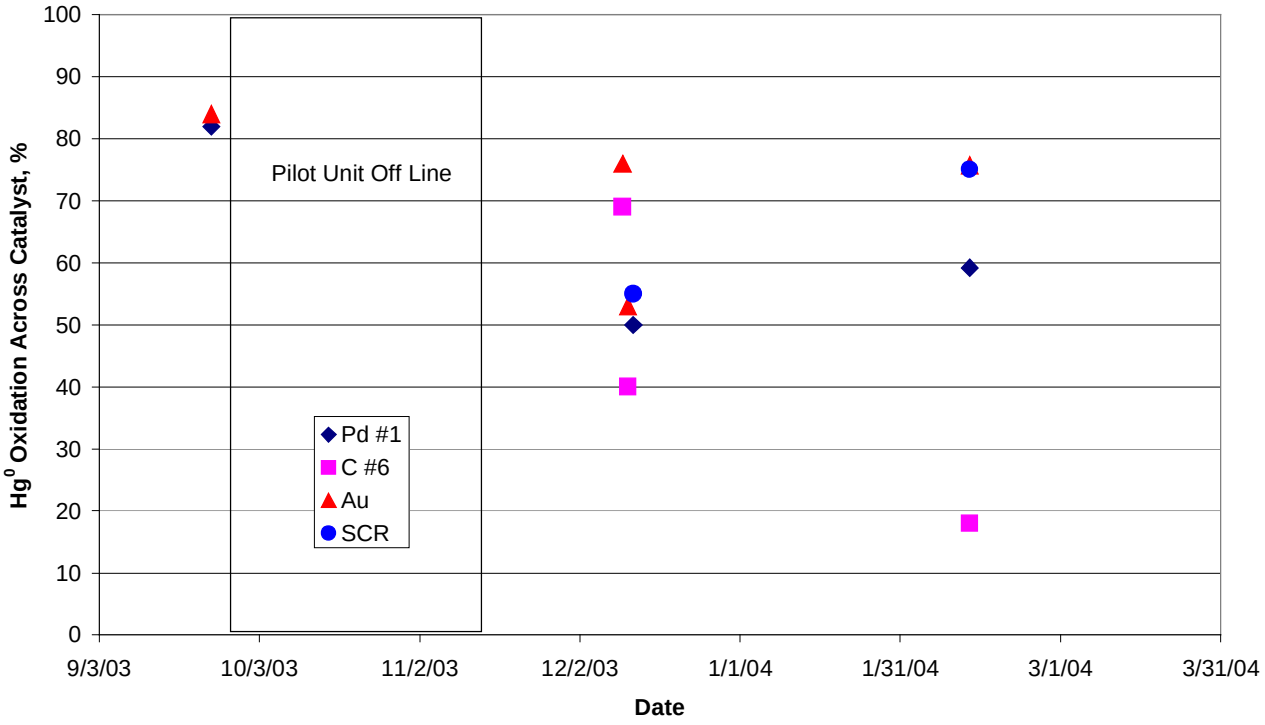

Figure 4. Catalyst Activity Versus Time in Service at Spruce Plant

Measurement of catalyst activity at Spruce is difficult for two reasons. One is that because of mercury oxidation and capture across the fabric filter, the elemental mercury concentrations at the oxidation catalyst pilot unit are relatively low, typically 3 µg/Nm^3 or lower. This means that for well performing catalysts, the catalyst outlet elemental mercury concentrations are on the order of 1 µg/Nm^3 or less, a low concentration that is difficult to measure accurately with the Hg SCEM (or by any other method).

The second difficulty is that the pilot inlet total and elemental mercury concentrations are observed to change significantly throughout the day, perhaps being impacted by factors such as fabric filter pressure drop and compartment cleaning cycles. Significant temporal variations were seen during both the December and February measurements. A single Hg SCEM is being used to quantify catalyst performance and must cycle between the pilot inlet flue gas sample and the catalyst chamber outlet samples, so inlet concentration variations can markedly impact observed mercury adsorption and elemental mercury oxidation percentages. As an example, on February 13, the pilot unit inlet elemental mercury concentration averaged 1.23 µg/Nm^3 at 10 a.m., but by

1:30 p.m. the average was 2.45 μg/Nm^3. This makes it difficult to determine what inlet value to use to calculate oxidation percentages for catalyst outlet elemental mercury concentrations measured between these two times. The latter value was used in Table 3 to calculate and report oxidation percentages across the catalysts.

Based on the February results, the Au and SCR catalysts are the most active, measured to be achieving approximately 75% elemental mercury oxidation. However, since the catalyst outlet mercury concentrations were less than 1 μg/Nm^3, this percentage cannot be stated with high precision. That is, we are unsure of the measurement precision at the value of 0.6 μg/Nm^3 seen at the outlets of these two catalysts. The Pd #1 catalyst is achieving approximately 60% oxidation, and the C #6 catalyst only 20% oxidation. It is interesting that the two better performing catalysts in the CCS pilot unit appear to be poorer performers at Spruce.

The Au catalyst oxidation measurement has been relatively consistent between the September, December and January, while the other catalysts have seen more varied results. The C #6 catalyst dropped in apparent oxidation percentage between December and January, while the Pd #1 and SCR catalysts increased. The next set of measurements will be made in April. It is hoped that with all new bags in the west fabric filter, the pilot inlet elemental mercury concentrations will be higher and more consistent, which should allow better measurement of catalyst performance.

Flue Gas Characterization Results

Also during the week of December 8, 2003, SCEM relative accuracy measurements were made using the Ontario Hydro method at the pilot unit inlet and the outlets of each of the four catalyst chambers. These results were not yet available for reporting in the previous quarter's Technical Progress Report, so they are presented and discussed here.

Metco Environmental collected the gas samples, and URS analyzed the resulting impinger solutions for mercury content. Tables 4 and 5 present the Ontario Hydro measurement results, along with average Hg SCEM data collected during the same time period as each Ontario Hydro run, for measurements on December 11 and 12, respectively.

The tables also show the relative accuracy between the Ontario Hydro and Hg SCEM measurements, calculated as the SCEM result minus the Ontario Hydro result, with the difference being expressed as a percentage of the Ontario Hydro result. In general, the total mercury concentration measurements agreed well between the two techniques, with the three-run average relative accuracy values ranging from –17% to +11%. The oxidized mercury concentration values also agree reasonably well between the two methods, with the average relative accuracy values ranging from –22% to +7%. This is seen as very good agreement, especially given the observed temporal concentration variations in the flue gas during this time period.

Table 4. Summary of Ontario Hydro Results from Spruce Plant, December 11, 2003

Sample Location	Ontario Hydro Results (corrected to 5% O_2)						Hg SCEM Results (corrected to 5% O_2)					Relative Accuracy		
	$\%O_2$ in Flue Gas	Hg Ox. ($\mu g/Nm^3$)	Hg^0 ($\mu g/Nm^3$)	Total Hg ($\mu g/Nm^3$)	Total Hg Oxidation (%)	Hg^0 Ox. Across Catalyst (%)	Hg Ox. ($\mu g/Nm^3$)	Hg^0 ($\mu g/Nm^3$)	Total Hg ($\mu g/Nm^3$)	Total Hg Oxidation (%)	Hg^0 Ox. Across Catalyst (%)	Hg Ox. (%)	Hg^0 (%)	Hg Total (%)
Pilot Inlet	5.8	11.3	1.14	12.4	91	-	9.53	1.17	10.7	89	-	-16	2	-14
	5.7	12.3	1.42	13.7	90	-	10.69	1.97	12.7	84	-	-13	39	-8
	5.8	8.76	0.63	9.39	93	-	8.57	1.43	10.0	86	-	-2	129	7
Average	**5.8**	**10.8**	**1.06**	**11.9**	**91**	**-**	**9.60**	**1.52**	**11.1**	**86**	**-**	**-11**	**43**	**-6**
C #6 Outlet	5.4	12.4	0.23	12.6	98	80	7.89	2.33	10.2	77	-99	-36	898	-19
	5.4	15.6	0.36	16.0	98	75	13.1	0.66	13.8	95	66	-16	85	-14
	5.8	9.52	0.34	9.86	97	46	14.5	0.52	15.1	97	64	53	55	53
Average	**5.5**	**12.5**	**0.31**	**12.8**	**98**	**71**	**11.8**	**1.17**	**13.0**	**91**	**23**	**-5**	**279**	**1**
Au Outlet	5.6	12.7	0.14	12.8	99	88	9.24	1.09	10.3	89	7	-27	675	-19
	5.6	13.6	0.45	14.0	97	68	10.8	1.22	12.0	90	38	-20	170	-14
	5.8	9.95	0.23	10.2	98	63	8.03	0.4	8.43	95	72	-19	73	-17
Average	**5.7**	**12.1**	**0.27**	**12.3**	**98**	**74**	**9.36**	**0.90**	**10.3**	**91**	**41**	**-22**	**229**	**-17**

Table 5. Summary of Ontario Hydro Results from Spruce Plant, December 12, 2003

Sample Location	Ontario Hydro Results (corrected to 5% O_2)						Hg SCEM Results (corrected to 5% O_2)					Relative Accuracy		
	$\%O_2$ in Flue Gas	Hg Ox. ($\mu g/Nm^3$)	Hg^0 ($\mu g/Nm^3$)	Total Hg ($\mu g/Nm^3$)	Total Hg Oxidation (%)	Hg^0 Ox. Across Catalyst (%)	Hg Ox. ($\mu g/Nm^3$)	Hg^0 ($\mu g/Nm^3$)	Total Hg ($\mu g/Nm^3$)	Total Hg Oxidation (%)	Hg^0 Ox. Across Catalyst (%)	Hg Ox. (%)	Hg^0 (%)	Hg Total (%)
Pilot Inlet	5.8	9.94	0.15	10.1	99	-	7.42	3.44	10.9	68	-	-25	2270	8
	5.4	10.6	0.46	11.0	96	-	8.65	1.27	9.92	87	-	-18	176	-10
	5.4	10.1	0.53	10.6	95	-	13.7	0.78	14.5	95	-	36	47	37
Average	**5.5**	**10.2**	**0.38**	**10.6**	**96**	**-**	**9.9**	**1.83**	**11.8**	**84**	**-**	**-3**	**383**	**11**
Pd #1 Outlet	6.0	11.2	0.35	11.5	97	-141	9.09	1.4	10.5	87	59	-19	300	-9
	6.0	12.3	0.36	12.7	97	22	9.97	0.69	10.7	94	46	-19	92	-16
	5.4	11.0	0.37	11.4	97	31	14.5	0.2	14.7	99	74	32	-45	30
Average	**5.8**	**11.5**	**0.36**	**11.9**	**97**	**5**	**11.2**	**0.76**	**12.0**	**94**	**58**	**-3**	**113**	**1**
SCR Outlet	6.0	14.1	0.25	14.3	98	-69	11.0	1.14	12.1	91	67	-22	365	-15
	5.6	12.0	0.30	12.3	98	34	13.5	0.68	14.2	95	46	12	124	15
	5.4	11.3	0.29	11.6	97	45	15.4	0.16	15.6	99	79	37	-46	35
Average	**5.7**	**12.5**	**0.28**	**12.8**	**98**	**26**	**13.3**	**0.66**	**14.0**	**95**	**64**	**7**	**135**	**9**

Although the reported Ontario Hydro and Hg SCEM data were collected over the same time periods, the Ontario Hydro measurements represent time averaged values over two hours, whereas the Hg SCEM values represent an average over only a subset of time during that two hours. A single Hg SCEM was cycled between the pilot inlet and two catalyst outlet sample locations, and between measuring total and elemental mercury during that two-hour period. Thus, there are only about 20 minutes of SCEM measurement data at each location and for each mercury species during the two-hour Ontario Hydro run.

The elemental mercury concentrations did not agree well between the two methods. The three-run average relative accuracy values ranged from +43% to +383%, indicating that the Hg SCEM average values were higher than the Ontario Hydro average values by as much as a factor of four. A similar effect was seen in the SCEM relative accuracy tests conducted at CCS last July.[6] The individual run data show an even wider range of discrepancy between the methods, with individual relative accuracy values ranging from –46% to +2270%. It is not clear which, if either of the two methods more accurately reflects the amount of elemental mercury in the flue gas at Spruce, particularly for the catalyst outlet locations.

The Ontario Hydro elemental mercury values appear to be biased low. For example, the pilot unit average inlet mercury oxidation was measured at 96% on December 12, with individual runs showing 95 to 99% oxidation. Such high oxidation percentages do not seem plausible for a PRB flue gas, even with a fabric filter upstream. Given the low inlet elemental mercury concentration measured by the Ontario Hydro Method on the 12th, it is not clear that the Pd #1 and SCR catalyst outlet. elemental mercury concentration and catalyst oxidation percentage values shown in Table 5 represent measurable results.

There are also anomalies in the Hg SCEM results. For the four catalyst outlet locations and the pilot inlet location on one of the two days, there was an observed continual lowering of the elemental mercury concentration from the first through the third run periods. For these five measurement sets, the elemental mercury concentration measured on the first run at a particular location ranged from being 3 to 7 times the value measured on the third run at that location. It does not seem plausible that elemental mercury concentrations would drop so markedly during the course of a day, and it seems more likely that these trends represent some type of undiagnosed measurement bias.

Additional flue gas characterization measurements were made that week, including trace metals (EPA Method 29) and halogen species (Method 26a) at the pilot unit inlet, and sulfuric acid concentrations upstream and downstream of two catalysts (Controlled Condensation System). The Method 29 non-mercury metals results are presented in Table 6, while the mercury results are presented in Table 7. The data in both tables are discussed below.

The three Method 26a runs each showed pilot unit inlet HCl concentrations of 0.8 ppmv (dry basis), and Cl_2 concentrations of <0.02 ppmv (dry basis). The Controlled Condensation System runs showed very low sulfuric acid vapor concentrations at the pilot inlet and at the outlets of the C #6 and Au catalysts. All measured values were less than 0.1 ppmv of H_2SO_4 (dry basis), which we consider to be the detection limit of the method.

Table 6. Method 29 Data from the Spruce Oxidation Catalyst Pilot Unit Inlet, 9 December 2003 (all values shown in ppbv (dry gas basis), particulate values shown as equivalent gas-phase concentration)

	Gas Phase Measurement Results				Particulate Phase Measurement Results			
Sample ID	**Run 1**	**Run 2**	**Run 3**	**Average**	**Run 1**	**Run 2**	**Run 3**	**Average**
Start Time	**9:05**	**11:40**	**14:15**	**-**	**9:05**	**11:40**	**14:15**	**-**
Aluminum	5,416	23,243	14,357	**14,339**	128	54	234	**139**
Antimony	<542	<536	<5,495	**<2,191**	ND	ND	ND	**ND**
Arsenic	ND	ND	ND	**ND**	ND	ND	ND	**ND**
Barium	<3,620	<3,586	<3,598	**<3,601**	0.72	1.2	46.0	**16.0**
Beryllium	ND	ND	ND	**ND**	ND	ND	ND	**ND**
Cadmium	<111	<110	<110	**<110**	0.03	<0.09	<0.09	**<0.07**
Calcium	<310,053	<307,159	<308,247	**<308,486**	126	92	71	**96.4**
Chromium	<478	<474	<475	**<475**	0.45	*	*	**-**
Cobalt	<2107	<209	<210	**<842**	<1.7	<1.8	<1.7	**<1.7**
Copper	<977	<969	<972	**<973**	0.37	*	*	**-**
Iron	ND	2645	4867	**3756**	9.4	7.7	24.2	**13.8**
Lead	<120	<119	<119	**<119**	<0.10	<0.10	<0.10	**<0.10**
Magnesium	<510,785	<506,414	<508,208	**<508,469**	<416	<426	<417	**<419**
Manganese	<678	<672	<675	**<675**	0.58	0.42	1.02	**0.67**
Molybdenum	ND	ND	ND	**ND**	0.11	0.00	0.02	**0.04**
Nickel	<1,693	<1,678	<1,684	**<1,685**	<1.4	<1.4	<1.4	**<1.4**
Potassium	-	-	-	**-**	<259	<265	<259	**<261**
Selenium	283	<312	<313	**<303**	ND	ND	ND	**ND**
Silver	<629	<78	<78	**<262**	ND	ND	ND	**ND**
Sodium	-	-	-	**-**	290	*	467	**-**
Thallium	ND	ND	ND	**ND**	ND	ND	ND	**ND**
Tin	1,883	2,074	12,074	**5,344**	<1.7	<1.7	<1.7	**<1.7**
Titanium	<2,594	<2,572	<2,581	**<2,582**	1.6	1.5	2.6	**1.9**
Vanadium	ND	ND	ND	**ND**	ND	ND	ND	**ND**
Zinc	1,747	1,694	38	**1,160**	6.0	0.6	*	**-**

*Value measured was less than field blank value

Table 7. Method 29 Mercury Data from the Spruce Oxidation Catalyst Pilot Unit Inlet, 9 December 2003 (all values shown in mg/Nm^3, particulate values shown as equivalent gas-phase concentration)

	Particulate Phase Hg, mg/Nm^3	M29 Gas Phase Hg, mg/Nm^3	SCEM Gas Phase Hg, mg/Nm^3	Relative Accuracy, %
Run 1	0.01	14.1	-	-
Run 2	0.00	13.0	12.7	-1.9
Run 3	0.01	12.8	9.91	-23
Average	0.01	13.5	-	-

As might be expected downstream of a well-performing baghouse, the metals concentration results in Table 6 show very little particulate-bound metals. Even in the vapor phase, metals concentrations were below measurable levels for all but aluminum, iron, tin and zinc.

The Method 29 mercury concentrations are compared to mercury SCEM values in Table 7. SCEM data were not available for the first Method 29 run. For the second run, the relative accuracy between the two methods was very good, with the SCEM value being 2% lower than the Method 29 value. For the third run, the difference was greater, with the SCEM value being 23% lower.

Another type of gas characterization effort was conducted at Spruce, the first week of January. In this effort, URS set up two Hg SCEMs on the host unit ductwork, one on the outlet duct of one wet FGD absorber and one alternated between the wet fabric filter outlet and inlet ducts. The objective of these measurements was to characterize the mercury oxidation and removal across the fabric filter with the existing, aged bags prior to the rebagging effort, which was to begin the next week. The FGD outlet measurements were made to confirm that the mercury being oxidized across the fabric filter was, in fact, being removed by the wet FGD system at high efficiency. At the same time, CPS had contracted with Metco Environmental to collect stack gas metals concentration data by Method 29, so this provided an opportunity to compare SCEM results to reference method results for flue gas mercury concentration.

The Hg SCEM results are summarized in Table 8. The results show very high oxidation percentages across the fabric filter, but less mercury removal across the wet FGD absorber than expected. Unfortunately, there are measurements we would like to have now that were not made. No elemental mercury numbers were measured for the FGD outlet on January 6. Since Method 29 does not speciate mercury, the engineer running that analyzer concentrated on measuring only total mercury numbers for comparison. On the 7th we changed the procedure to measure both total and elemental mercury concentrations with the FGD outlet analyzer. Since only two analyzers were used, we had to get fabric filter outlet data one day and the inlet the other, so there are unfortunately no fabric filter outlet speciation data for the 7th, the only day we have FGD outlet speciation.

Table 8. Mercury Concentration Data at Spruce by Hg SCEM, January 2004

Location	Hg Conc. (μg/Nm³)			Overall Hg Ox. (%)	Hg Removal Relative to Fabric Filter Inlet (%)	Hg Ox. Across Fabric Filter (%)	Hg Removal Across FGD Absorber (%)		
	Hg Total	Hg^0	Hg^{+2}				Hg Total	Hg^0	Hg^{+2}
Fabric Filter Inlet (7-Jan)	13.5	11.3	2.19	16	-	-	-	-	-
Fabric Filter Outlet (6-Jan)	12.7	0.76	12.0	94	6*	93*	-	-	-
FGD Outlet (6-Jan)	4.78	-	-	-	65*	-	62		
FGD Outlet (7-Jan)	3.22	1.35	1.87	58	76	-	75*	-78*	84*

*Percentage was calculated using inlet data from the previous day, so the value shown may be biased by day-to-day variability

Consequently, we do not have a direct measure of elemental mercury "re-emissions" across the scrubber. Based on the fabric filter outlet for January 6 and the scrubber outlet for January 7, a

small amount of elemental mercury re-emissions is evident. However, since the two measurements are on different days and given the amount of variation previously and subsequently measured in elemental mercury concentrations at this site, this cannot be considered conclusive evidence of mercury re-emissions. Re-emissions are typically not seen across highly oxidized (low liquid-phase sulfite concentration) FGD absorbers. The FGD system at Spruce is not forced oxidized, but typically produces a highly oxidized gypsum byproduct due to high natural oxidation. During these flue gas measurements, URS collected FGD samples and analyzed FGD liquors for sulfite concentration. These analyses showed that the FGD byproduct solids were 100% oxidized to the sulfate (gypsum) form. However, in the liquid phase samples, individual sample sulfite concentrations were measured to range from 1.1 to 2.7 m-mol/l, with an average of 1.9 m-mol/l. Based on the results of bench-scale testing conducted by URS for team member EPRI, we would expect a small amount of mercury re-emissions at FGD liquor sulfite concentrations above 1 m-mol/l.[7]

The data in Table 8 show 84% removal of oxidized mercury across the FGD absorber, although this percentage is based on an FGD inlet oxidized mercury concentration measured the day before. This is a lower oxidized removal percentage than would be expected. The SO_2 removal across the FGD system at Spruce was about 92% at the time. One would expect similar or greater removal percentages for oxidized mercury, in the absence of re-emissions. The FGD inlet oxidized mercury concentration to the FGD system would have had to be greater than 20 $\mu g/Nm^3$ on January 7 for the actual oxidized mercury removal across the FGD absorber to have been above 90%, and such a concentration is above the range previously measured at Spruce. This consideration further supports the observation that there was a small amount of mercury re-emissions across the FGD system at Spruce.

CPS also reported the stack total mercury concentrations measured at Spruce by Metco Environmental by Method 29 (M29). For January 7, the stack and FGD outlet total mercury concentrations measured by SCEM should have been equivalent, as the FGD system was being operated with no flue gas bypass. On January 6, the FGD bypass damper was partially open and about 15 to 20% of the fabric filter outlet gas was bypassing the FGD system. Thus, the M29 results for the stack gas should have shown higher total mercury concentrations than the Hg SCEM results at the fabric filter outlet. A comparison between the two sets of measurements is shown in Table 9.

Table 9. Comparison of Stack M29 Data and Hg SCEM Data from FGD Outlet

Date	Time	Run	M29 Results at Stack (Hg, $\mu g/Nm^3$ @ 5% O_2)	Hg SECM Results at FGD Outlet (Hg, $\mu g/Nm^3$ @ 5% O_2)	Stack Temp (^oF)	Hg SCEM Results at FF Outlet (Hg, $\mu g/Nm^3$ @ 5% O_2)	Estimated % Gas Bypass	Estimated Hg SCEM Results at Stack (Hg, $\mu g/Nm^3$ @ 5% O_2)
6-Jan	0945-1205	1	<5.21	4.61	153	13.3	14%	5.9
6-Jan	1323-1546	2	<4.38	5.70	156	13.2	16%	6.9
Average			**<4.79**	**5.15**	**155**	**13.3**	**15%**	**6.4**
7-Jan	0800-1022	1	<2.59	4.22	132	-	0%	-
7-Jan	1110-1336	2	<3.51	2.28	133	-	0%	-
7-Jan	1430-1650	3	<2.96	2.10	134	-	0%	-
Average			**<3.02**	**2.87**	**134**	**-**	**0%**	**-**

The four columns of information on the right in Table 9 were used to estimate the stack total mercury concentration from the Hg SCEM data. SCEM data from the fabric filter outlet and FGD outlet were weighted based on a bypass percentage estimated from stack flue gas temperature.

The ability to compare the results of these two methods is confounded by the relatively high detection limits for mercury shown in the M29 results. The comparison in the table shows that the M29 measurements on January 6 were lower (based on the reported gas detection limits) than were measured with the SCEM, while the data from January 7 show reasonable agreement (assuming the actual M29 values were near the reported gas detection limits).

Since the Hg SCEM measurements were on the west side of the plant while the stack measurements were conducted on the mixed gas from both sides of the plant, it is possible that the mercury concentrations in the flue gas exiting the east fabric filter were lower than those from the west, leading to the observed differences measured by the two methods in different locations. On January 7, since all of the flue gas was scrubbed, oxidized mercury removal across the FGD absorbers would tend to minimize the effects of any side-to-side biases.

Laboratory Evaluation of Candidate Catalysts

No laboratory evaluations were conducted during the current quarter.

CONCLUSION

Because of the observed ash accumulation on the catalysts at CCS, provisions had to be made to help keep catalyst surfaces cleaner. Sonic horns are commonly used to clean catalysts on line in utility SCR applications for NO_X control, and appear to be similarly effective in this application (lower dust loading but horizontal gas flow). In 10 months of operation, the horns have been apparently been effective at limiting fly ash buildup in three of the four catalysts. For the fourth catalyst, SBA #5, it appears that either the sonic energy from the horn has not been sufficient to prevent fly ash accumulation, or that the sonic energy has caused damage to the honeycomb structure. Either could explain the increased pressure drop (and loss of activity) across this catalyst.

Catalyst activity measurements in February indicate that the horns have also been effective in maintaining catalyst activity for two more active catalyst materials. After 18 months of operation, the Pd #1 catalyst has seen some long-term loss in activity for elemental mercury oxidation, from slightly greater than 90% to between 65 and 70%. After 10 months of operation, the C #6 catalyst has dropped from greater than 95% to between 85 and 90%. The SCR catalyst has seen a more significant loss, dropping from 67% to less than 30% oxidation over an 18-month period (as measured by SCEM). The SBA #5 catalyst has dropped from 75% oxidation to about 30% oxidation over a 16-month period (also based on SCEM results). However, for the Pd #1, SCR and SBA #5 catalysts, these results are confounded by the fly ash buildup experienced prior to the sonic horn installations. The fly ash buildup could have had beneficial or negative effects on catalyst activity. If the catalysts can be deactivated by species in the flue gas, the honeycomb cells that were blocked by fly ash buildup may have been "protected" from deactivation by flue gas species. Conversely, the fly ash buildup could have directly affected catalyst activity in an adverse manner through physical blockage or chemical reactions at active sites. The C #6 has operated with a sonic horn in service to prevent fly ash buildup for its entire 10-months of operation. Continued operating time on all four catalysts will better quantify activity loss over time with the sonic horns in service to limit fly ash buildup.

At the Spruce site, the fabric filter upstream of the pilot unit has had two implications on the pilot testing. One is that is does not appear that sonic horns will be required to keep fly ash from accumulating within the catalyst cells. The other implication is that the fabric filter oxidizes a high percentage of the elemental mercury in the air heater outlet flue gas, so the inlet gas to the pilot unit contains relatively low elemental mercury concentrations (typically 1 to 3 $\mu g/Nm^3$). This makes evaluation of catalyst performance difficult, as it is difficult to quantify flue gas elemental mercury concentrations below 1 $\mu g/Nm^3$. It is hoped that after the fabric filter bag replacement was completed in February/March 2004, pilot unit inlet elemental mercury concentrations will increase to a higher concentration that will better support oxidation catalyst evaluation.

Based on results to date, the Au and SCR catalysts are the most active in this flue gas, achieving about 75% elemental mercury oxidation, with the Pd #1 catalyst being somewhat less active at 60% oxidation. The activity of the C #6 is much lower, at about 20% elemental mercury oxidation. This is a surprising result, since the C #6 catalyst has been very active at CCS. During the upcoming quarter, site measurements will be conducted to determine whether this catalyst

has, in fact, deactivated or whether the low activity result represents a measurement error due to the low catalyst inlet elemental mercury concentrations in the flue gas. Low, and variable elemental mercury concentrations in the flue gas at the Spruce pilot unit inlet confound the ability to accurately measure catalyst activity.

There continues to be a bias between Ontario Hydro and Hg SCEM results for elemental mercury concentration in the flue gas downstream of the catalysts. Such biases were seen in CCS results from July 03 and Spruce results from December 03. It is not clear whether the bias is in the Ontario Hydro method, the Hg SCEM method, or both. All three available methods (Hg SCEM, Ontario Hydro, and Method 29) show reasonable agreement for measuring total mercury concentrations.

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